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Indian Standard

METHODS OF CHEMICAL
ANALYSIS OF METALLIC SILICON

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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METHODS OF CHEMICAL ANALYSIS OF METALLIC SILICON

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CONTENTS

	PAGE
0. FOREWORD	4
1. SCOPE	5
2. SAMPLING	5
3. QUALITY OF REAGENTS	5
4. GENERAL	5
5. DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD ...	5
6. DETERMINATION OF ALUMINIUM BY THE OXINE METHOD ...	7
7. DETERMINATION OF IRON BY THE POTASSIUM THIOCYANATE (COLORIMETRIC) METHOD	9
8. DETERMINATION OF CALCIUM BY THE OXALATE (VOLUMETRIC) METHOD	10
9. DETERMINATION OF PHOSPHORUS BY THE MOLYBDATE (ALKALIMETRIC) METHOD	11
10. DETERMINATION OF SULPHUR BY THE GRAVIMETRIC METHOD	14
11. DETERMINATION OF CARBON BY THE DIRECT COMBUSTION (GRAVIMETRIC) METHOD	15

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF METALLIC SILICON

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 4 December 1964, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 For the analysis of metallic silicon, used mostly in the production of steel, various laboratories in the country employ different methods; the results thus reported are not always comparable. Necessity was, therefore, felt for prescribing standard methods which can primarily serve as referee methods and could also be used by the laboratories for their day-to-day work.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

1962 Book of ASTM methods for chemical analysis of metals. American Society for Testing and Materials.

FURMAN (N H) AND SCOTT (W W), Ed. Standard methods of chemical analysis. 1962. D. Van Nostrand Company Inc., New York.

LUNDELL (G E F), HOFFMAN (J I) AND BRIGHT (M A). Chemical analysis of iron and steel. 1946. 4th Printing. John Wiley & Sons, New York.

PIGGOT (E C). Ferrous analysis—modern practice and theory, Ed 2. 1954. Chapman & Hall, London.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard covers methods of chemical analysis of metallic silicon of four grades as specified in IS : 2301-1963*.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS : 1472 (Part II) - 1962†.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960‡) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. GENERAL

4.1 Use of Filter Papers — In some of the methods given in this standard, numbers of Whatman filter papers which are commonly used, have been mentioned. However, any other suitable brand of filter paper of corresponding porosity may also be used.

5. DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD

5.1 Outline of the Method — The sample is decomposed by fusion with sodium peroxide in a nickel crucible, acidified with hydrochloric acid and dehydrated. Silica is filtered and determined by hydrofluorization.

5.2 Reagents

5.2.1 Fusion Mixture — Mix 6 g of anhydrous sodium carbonate with 4 g of anhydrous potassium carbonate in a glass mortar.

5.2.2 Sodium Peroxide — solid.

5.2.3 Concentrated Hydrochloric Acid — sp gr 1.16 (conforming to IS : 265-1962§).

5.2.4 Dilute Hydrochloric Acid — 1 : 50 (v/v).

5.2.5 Perchloric Acid — 70 percent.

5.2.6 Concentrated Sulphuric Acid — sp gr 1.84 (conforming to IS : 266-1961||).

*Specification for metallic silicon.

†Methods of sampling ferro alloys, Part II.

‡Specification for water, distilled quality (*revised*).

§Specification for hydrochloric acid (*revised*).

||Specification for sulphuric acid (*revised*).

5.2.7 Hydrofluoric Acid — 40 percent.

5.2.8 Sodium Carbonate — anhydrous.

5.3 Procedure

5.3.1 Transfer 0.5 g of an accurately weighed sample to a 30-ml nickel crucible. Add 3 g of fusion mixture and approximately 8 g of dry sodium peroxide, and mix the contents thoroughly with a small nickel rod. Cover the mixture with a layer of another 2 g of dry sodium peroxide.

5.3.2 Fuse the contents of the crucible carefully over the flame of a burner. The fusion is best accomplished by holding the crucible with a pair of tongs and slowly revolving it around the outer edge of the flame until the contents have melted down quietly, care being taken not to raise the temperature so rapidly as to cause spattering. When the fusion is complete, impart a slight rotary motion to the crucible to stir up any unattacked particles of alloy on the bottom or sides of the crucible and maintain the contents at a low red heat. Just before completion of the fusion, which only requires three or four minutes, increase the temperature to bright redness for a minute. When the tightly covered crucible has cooled down for a sufficiently long time, but has not reached room temperature, tap it on an iron plate several times to loosen the fused mass from the crucible. Allow the melt to cool.

5.3.3 Transfer the cold melt to a 15-cm covered porcelain basin to which 50 ml of cold water is added cautiously. As soon as the reaction is over, allow the basin and its contents to cool somewhat and add 50 ml of concentrated hydrochloric acid, using more if necessary, to render the solution acidic. Evaporate the solution to dryness on a sand- or water-bath. Then heat for 30 minutes at a temperature of about 110°C. More prolonged heating or heating at higher temperature will render the iron oxide more insoluble. Allow the basin to cool, and add 10 ml of concentrated hydrochloric acid and heat for 5 minutes or so, breaking up any hard lump with a glass rod. Add 150 ml of water and shake the solution just sufficiently long to dissolve most of the sodium chloride; filter through filter paper No. 40 and wash 5 to 6 times with dilute hydrochloric acid and then thoroughly with hot water. Preserve the residue (R_1) and the filtrate.

5.3.4 Add 30 ml of perchloric acid to the combined filtrate and washings as obtained under **5.3.3**; evaporate and fume strongly for 5 to 10 minutes. Allow to cool somewhat, redissolve in 40 ml of concentrated hydrochloric acid and 150 ml of water and filter through filter paper No. 40 detaching adhering silica by means of rubber tipped glass rod and wash with hot water. Preserve the residue (R_2) and the filtrate.

5.3.5 Ignite the two residues (R_1) and (R_2) in a platinum crucible first at a moderate heat with access of air and finally at 950°C for 30 minutes or longer. Weigh the crucible. Moisten the ignited residue with water and treat it with 3 to 4 drops of concentrated sulphuric acid and 5 to 10 ml of hydrofluoric acid and cautiously evaporate to dryness and then heat strongly for 5 minutes. Preserve the residue.

5.3.6 Carry out a blank determination following the same procedure and using the same amount of all reagents but without the sample.

5.3.7 Fuse the residue preserved under **5.3.5** with sodium carbonate. Extract the melt with 10 to 15 ml of concentrated hydrochloric acid and mix with the filtrate preserved under **5.3.4**. This solution should be used for determination of aluminium, iron and calcium.

5.4 Calculation

$$\text{Silicon, percent} = \frac{(A - B) \times 46.72}{C} + D$$

where

A = weight in g of silica obtained in the test,

B = weight in g of the silica obtained in the blank,

C = weight in g of the sample taken, and

D = percent of silicon recovered in **6.3.3**.

6. DETERMINATION OF ALUMINIUM BY THE OXINE METHOD

6.1 Outline of the Method — In the filtrate from **5.3.7**, mixed oxides are precipitated by ammonium hydroxide. The precipitate is filtered, washed and dissolved in hydrochloric acid and iron separated. Alumina is precipitated from acetate solution as oxinate filtered, dried and weighed.

6.2 Reagents

6.2.1 Concentrated Ammonium Hydroxide — 20 percent.

6.2.2 Ammonium Chloride — solid.

6.2.3 Methyl Red Indicator Solution — 0.4 g per litre. Dissolve 0.1 g of methyl red in 3.72 ml of sodium hydroxide solution (0.1 N) and dilute 250 ml with water. Filter if necessary.

6.2.4 Ammonium Chloride Wash Solution — Dilute 25 ml of hydrochloric acid, neutralize to methyl red with concentrated ammonium hydroxide, and dilute to one litre. Add 2 drops of concentrated ammonium hydroxide in excess.

6.2.5 Concentrated Hydrochloric Acid — See **5.2.3**.

6.2.6 Sodium Potassium Tartarate Solution — 20 percent (w/v).

6.2.7 Hydrogen Sulphide — gas.

6.2.8 Ammonium Sulphide Wash Solution — Add 5 g of ammonium chloride to ammonium sulphide solution (5 percent).

6.2.9 Bromocresol Purple Indicator Solution — Warm 0.100 g of bromocresol purple with 5 ml of alcohol (90 percent) until dissolved. Add 100 ml of alcohol (20 percent), 3.7 ml of (exactly 0.05 N) sodium hydroxide solution and dilute with alcohol to make up to 250 ml.

6.2.10 Dilute Ammonium Hydroxide — 1 : 3 (v/v).

6.2.11 'Oxine' Solution — 2 percent (w/v). Dissolve 5 g of 8-hydroxyquinoline in 15 ml of glacial acetic acid by warming and dilute with water to 250 ml.

6.2.12 Ammonium Acetate — solid.

6.3 Procedure

6.3.1 Boil the filtrate from **5.3.7** and precipitate mixed oxides by adding concentrated ammonium hydroxide in presence of ammonium chloride, using methyl red as indicator until the colour changes to distinct yellow. Allow to settle, filter and wash four times with hot ammonium chloride wash solution. Preserve the filtrate and washings.

6.3.2 Return the precipitate to the beaker and dissolve by heating with 10 ml of concentrated hydrochloric acid and some water. Dilute, heat to boiling and precipitate as before. Filter the precipitate and wash with hot ammonium chloride wash solution. Preserve the residue of oxides of iron and aluminium. Mix the filtrate with the filtrate and washings preserved under **6.3.1**. Preserve the combined filtrates for the determination of calcium (see **8**).

6.3.3 Ignite the residue preserved under **6.3.2** at 950°C in a platinum crucible and weigh. Fuse the ignited residue with potassium bisulphate. Take up the melt with 150 ml of water and 5 ml of concentrated sulphuric acid. Filter out the silica. Ignite silica as usual and determine the percentage of silica. Add the figure to percent of silicon obtained under **5.4**.

6.3.4 Pass hydrogen sulphide gas through the filtrate obtained under **6.3.3** to remove traces of platinum and filter if any precipitate appears. Add, to the filtrate, one gram of tartaric acid and pass hydrogen sulphide again for 5 to 10 minutes after making the solution ammoniacal. Allow to stand for 30 minutes and filter off ferrous sulphide, washing with ammonium sulphide wash solution. Preserve the precipitate for the determination of iron (see **7**).

6.3.5 Acidify the filtrate as obtained under **6.3.4** with concentrated hydrochloric acid and boil the solution to remove hydrogen sulphide. Filter the precipitated sulphur through No. 551 filter paper and wash 6 to 7 times with hot water. Concentrate the solution to 200 ml, add 3 to 4 drops of bromocresol purple indicator solution and little concentrated ammonium hydroxide slowly until the colour just turns violet. Allow the solution to cool to 70°C to 80°C and add 25 to 30 ml of oxine solution, followed by 7 to 8 g of ammonium acetate slowly with stirring, and maintain the solution between 70° to 80°C until the precipitate has coagulated. Filter the precipitate into dried and weighed sintered (No. 3 Porosity) Gooch crucible and wash the precipitate with hot water 10 to 12 times. Dry the precipitate at 130°C in an air-oven to constant weight and weigh it as $\text{Al}(\text{C}_9\text{H}_6\text{ON})_3$.

6.4 Calculation

$$\text{Aluminium, percent} = \frac{A \times 5.872}{B}$$

where

A = weight in g of aluminium oxinate, and

B = weight in g of sample represented by the solution taken.

7. DETERMINATION OF IRON BY THE POTASSIUM THIOCYANATE (COLORIMETRIC) METHOD

7.1 Outline of the Method — The red colour, produced by potassium thiocyanate in the iron solution obtained from **6.3.4** is compared with that produced under similar conditions in standard iron solution containing a known amount of iron oxide.

7.2 Reagents

7.2.1 Dilute Hydrochloric Acid — 1 : 3 (v/v).

7.2.2 Potassium Chlorate — solid.

7.2.3 Standard Iron Solution — (1 ml = 0.0001 g of Fe). Dissolve 0.7025 g of ferrous ammonium sulphate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water, add 5 ml of concentrated sulphuric acid (sp gr 1.84) and oxidize with bromine water, boil to expel bromine, cool and make up the volume to one litre.

7.2.4 Potassium Thiocyanate Solution — Dissolve 97 g of potassium thiocyanate (KCNS) in water and dilute to one litre.

7.3 Procedure

7.3.1 Dissolve the precipitate of ferrous sulphide as obtained under **6.3.4** in dilute hydrochloric acid containing a pinch of potassium

chlorate. Wash the paper thoroughly and boil out chlorine from the filtrate.

7.3.2 Pour the solution after cooling in a 250-ml volumetric flask and make up the volume to the mark with cold water. Dilute an aliquot of this solution to 250 ml and use this as test solution.

7.3.3 Dilute 5 ml of standard iron solution to 100 ml and transfer to a burette reading to 0.1 ml; fill a similar burette with water. Fill two colorimeter test cells; one with 10 ml of potassium thiocyanate solution together with 10 ml of test solution and the other with 10 ml of potassium thiocyanate solution and 10 ml of water. Add the dilute standard iron solution from the burette to the latter and an equivalent amount of water from the other burette to the former stirring thoroughly until the tints in the two cells are approximately same. Examine against a neutral background by light transmitted through the sides of the cells or compare in a suitable colorimeter.

7.4 Calculation

$$\text{Iron, percent} = A \times \frac{H_1}{H_2} \times \frac{250}{B} \times \frac{0.0001 \times 100}{C}$$

where

A = volume in ml of dilute standard iron solution,

H_1 = height of column of dilute standard iron solution,

H_2 = height of column of the test solution,

B = volume in ml of aliquot of the test solution used, and

C = weight in g of the sample represented by the aliquot taken.

8. DETERMINATION OF CALCIUM BY THE OXALATE (VOLUMETRIC) METHOD

8.1 Outline of the Method — In the ammoniacal filtrate obtained in 6.3.2, nickel is precipitated as sulphide, and is filtered off. Calcium in the filtrate is precipitated as oxalate, filtered, washed dissolved in hot dilute sulphuric acid and titrated with standard potassium permanganate solution.

8.2 Reagents

8.2.1 Hydrogen Sulphide — gas.

8.2.2 Concentrated Ammonium Hydroxide — 20 percent.

8.2.3 Ammonium Oxalate — solid.

8.2.4 Dilute Sulphuric Acid — 1:4 (v/v).

8.2.5 Standard Potassium Permanganate Solution (0.01 N) — Dissolve 0.32 g of pure potassium permanganate in one litre of water in a dark glass-stoppered bottle and standardize against oxalic acid (AR).

8.3 Procedure

8.3.1 Boil the filtrate as obtained under 6.3.2 and pass hydrogen sulphide for a sufficient time in the hot alkaline solution to precipitate out all nickel as nickel sulphide which is indicated by the solution becoming colourless. Filter the solution. If the solution shows cloudiness or a tinge of colour, add little more concentrated ammonium hydroxide to make it strongly alkaline and boil again, pass hydrogen sulphide as before and filter. Boil this colourless filtrate to remove all hydrogen sulphide and to reduce the volume to 100 ml. Add slight excess of concentrated ammonium hydroxide and about 0.5 g of ammonium oxalate. Let the contents boil for 5 minutes, cool, filter and wash the precipitate with small portion of hot water till free from oxalate.

8.3.2 Puncture the paper and wash as much as possible of the precipitate with small portion of hot water into the beaker in which the precipitate was made. Pour about 50 ml of hot dilute sulphuric acid through the filter, wash thoroughly. Heat to 75° to 80°C and titrate with standard permanganate solution.

8.4 Calculation

$$\text{Calcium, percent} = \frac{A \times B \times 0.020}{C} \times 100$$

where

A = volume in ml of the standard permanganate solution required for titration,

B = normality of the standard permanganate solution, and

C = weight in g of the sample represented by the solution taken.

9. DETERMINATION OF PHOSPHORUS BY THE MOLYBDATE (ALKALIMETRIC) METHOD

9.1 Outline of the Method — The sample is dissolved in an acid mixture of hydrofluoric acid and nitric acid. After removal of hydrofluoric acid, by fuming with perchloric acid, phosphorus is precipitated as phosphomolybdate and estimated by dissolving the precipitate in a known excess of sodium hydroxide and titrating the excess of alkali against standard hydrochloric acid.

9.2 Reagents

9.2.1 Concentrated Nitric Acid — sp gr 1.42 (conforming to IS:264-1950*).

*Specification for nitric acid.

9.2.2 Hydrofluoric Acid — 40 percent.

9.2.3 Sodium Nitrate — solid.

9.2.4 Concentrated Sulphuric Acid — See 5.2.6.

9.2.5 Concentrated Hydrochloric Acid — See 5.2.3.

9.2.6 Dilute Hydrochloric Acid — 1 : 1 (v/v).

9.2.7 Sodium Carbonate — anhydrous.

9.2.8 Potassium Permanganate Solution — 2.5 percent (w/v).

9.2.9 Potassium Nitrite Solution — 10 percent (w/v).

9.2.10 Ammonium Molybdate Solution — Add solution (A) (see 9.2.10.1) slowly and with constant stirring to solution (B) (see 9.2.10.2) kept cool in a cold water-bath. Add 10 ml of ammonium phosphate (one gram per litre) and keep the solution at least for 24 hours. Filter the solution through filter paper No. 1 before use.

9.2.10.1 Solution (A) — Dissolve 100 g of molybdic acid (M_6O_3 , 80 percent) or 118 g of ammonium molybdate in a mixture of 145 ml of concentrated ammonium hydroxide and 270 ml of water. Cool the solution.

9.2.10.2 Solution (B) — Add 490 ml of concentrated nitric acid to 1 150 ml of water and cool.

9.2.11 Dilute Nitric Acid — 2 : 98 (v/v).

9.2.12 Potassium Nitrate Wash Solution — one percent.

9.2.13 Phenolphthalein Indicator Solution — 0.2 percent (w/v) in rectified spirit.

9.2.14 Sodium Hydroxide Solution — 0.1 N. Dissolve 4.5 g of purified sodium hydroxide in one litre of freshly boiled and cooled water, add a slight excess of barium hydroxide solution, let stand for 24 hours and decant the liquid before use.

9.2.15 Standard Hydrochloric Acid Solution — 0.1 N. Dilute 11 ml of concentrated hydrochloric acid to one litre and standardize the solution against sodium carbonate.

9.3 Procedure

9.3.1 Transfer 2 g of an accurately weighed sample into a 300-ml platinum dish. Add 30 ml of concentrated nitric acid. Add 2 ml of hydrofluoric acid, warm until violent reaction starts; stop heating and add hydrofluoric acid drop-wise from time to time until the sample is

almost dissolved. Add 3 g of sodium nitrate. Evaporate to small volume. Add again 10 ml of concentrated nitric acid and 5 ml of hydrofluoric acid, evaporate to dryness. Add 10 ml of concentrated sulphuric acid and evaporate to fumes. Add 100 ml of warm water and 10 ml of concentrated hydrochloric acid. Boil to dissolve the salts. Filter through filter paper No. 42. Wash the residue on the filter paper with dilute hydrochloric acid and hot water. Preserve the filtrate if there is any undissolved sample, weighing more than 0.05 g, the procedure shall be started afresh.

9.3.2 Ignite the filter paper with the silica residue in a platinum crucible and fuse it with sodium carbonate (seven times the weight of the residue). Extract the fused mass with 15 ml of concentrated hydrochloric acid and transfer into a 300-ml porcelain basin with cover. Wash the adhering particles with hot water in the crucible into the porcelain basin. Evaporate the solution and bake twice adding 3 ml of concentrated hydrochloric acid, each time.

9.3.3 Dissolve the salt with 25 ml of dilute hydrochloric acid and dilute with hot water to 50 ml. Filter through a filter paper No. 42. Discard the residue and mix the filtrate with the filtrate preserved under 9.3.1.

9.3.4 Concentrate the solution to 150 ml by boiling. Add potassium permanganate solution till the manganese dioxide is precipitated. Dissolve the precipitate with a few drops of potassium nitrite solution. Boil thoroughly and cool. Add concentrated ammonium hydroxide in slight excess. Neutralize with concentrated nitric acid and add 2 to 3 ml in excess. Adjust the temperature of the solution to 70° to 80°C and add 40 ml of ammonium molybdate solution. Shake vigorously and allow to stay for one hour or so to settle down the precipitate.

9.3.5 Filter the yellow ammonium phosphomolybdate precipitate through filter paper pulp and wash with cold dilute nitric acid solution (2 : 98).

9.3.6 Wash three to four times with cold dilute nitric acid. Wash with potassium nitrate wash solution until 10 ml of the filtrate collected in a test tube do not consume more than one drop of the sodium hydroxide solution using phenolphthalein indicator, showing the filtrate to be free of acid. When the washing is complete, transfer the filter paper pulp along with the precipitate to the original beaker in which the precipitation was done. Add with stirring a measured excess of sodium hydroxide solution until all the yellow precipitate has dissolved and the solution is colourless. Add 50 to 60 ml of water and about five millilitres of sodium hydroxide solution in excess and stir. Add three to four drops of phenolphthalein indicator solution and titrate with the standard

hydrochloric acid solution, until the pink colour is completely discharged.

9.3.7 Carry out a blank on the same quantity of sodium hydroxide solution which was added in the test, by titrating it with the standard hydrochloric acid solution, using three to four drops of phenolphthalein indicator solution.

9.3.8 Calculation

$$\text{Phosphorus, percent} = \frac{(A-B)C \times 0.135}{D}$$

where

A = volume in ml of the standard hydrochloric acid solution required for the blank test,

B = volume in ml of the standard hydrochloric acid solution required to titrate the excess,

C = normality of the standard hydrochloric acid solution, and

D = weight in g of the sample taken.

10. DETERMINATION OF SULPHUR BY THE GRAVIMETRIC METHOD

10.1 Outline of the Method — After the removal of silica, sulphur is precipitated as barium sulphate by means of barium chloride in acid medium filtered, ignited and weighed as BaSO_4 .

10.2 Reagents

10.2.1 Barium Chloride Solution — 10 percent (*w/v*).

10.2.2 Hydrochloric Acid Wash Solution — Dilute 5 ml of concentrated hydrochloric acid with water to 1000 ml and add one gram of barium chloride.

10.3 Procedure

10.3.1 Dissolve 2 g of an accurately weighed sample in nitric acid and remove silica as under **9.3.1** to **9.3.3**.

10.3.2 Concentrate the filtrate to adjust the acidity to 2 percent by volume of hydrochloric acid. Warm it to 60° to 70°C and add 10 ml of barium chloride solution dropwise with constant stirring. Let the solution stand for two hours at steam-bath and overnight at the room temperature. Filter through filter paper No. 42, washing the precipitate from the beaker with a jet of cold hydrochloric acid wash solution. Wash until the filtrate is free from iron salts, and continue washing with cold water until chlorides are removed. Transfer the pad and precipitate to a platinum or porcelain crucible.

10.3.3 Dry the filter pad completely at a low temperature (in the mouth of a muffle furnace) until the paper chars and gently burns; ignite finally at a temperature of 800° to 900°C to constant weight.

10.3.4 Carry out a blank determination following the same procedure and using the same quantity of all reagents but without the sample.

10.4 Calculation

$$\text{Sulphur, percent} = \frac{(A - B) \times 13.74}{C}$$

where

A = weight in g of the barium sulphate obtained in **10.3.3**,

B = weight in g of barium sulphate obtained in the blank determination, and

C = weight in g of the sample taken.

11. DETERMINATION OF CARBON BY THE DIRECT COMBUSTION (GRAVIMETRIC) METHOD

11.1 Outline of the Method— The sample is burnt in a stream of pure oxygen, and the resulting carbon dioxide is absorbed in soda asbestos after removal of the contaminants. The increase in weight of the absorbent is proportional to the carbon content of the sample.

11.2 Apparatus— The assembly of apparatus is shown in Fig. 1.

11.2.1 Source of Oxygen Supply— gas holder A (capacity 8 to 10 litres) containing 99.5 percent oxygen, free from carbonaceous matter, connected by rubber tubing with the gas purifying train. The gas holder should be filled up before starting the operation.

11.2.2 Oxygen Purification Train— consists of Arnold bottles (B) and (C) the latter containing concentrated sulphuric acid (*see* 5.2.6), the exit bulb of which is packed with glass wool and another absorption bottle (D) containing soda asbestos which passes through IS Sieve 140 but is retained on IS Sieve 50 and 'anhydrone' in layers, connected in series between the gas holder and the inlet end of the combustion tube. A layer of about 12 mm of anhydrone is placed on top for removing moisture in the gas and the soda asbestos removes carbon dioxide in the gas.

11.2.3 Furnace— a horizontal tube furnace (E) heated to 1350°C by electricity.

11.2.4 Combustion Tube— A combustion tube approximately 650 mm long and of 20 mm inner diameter made of fused silica, fireclay or

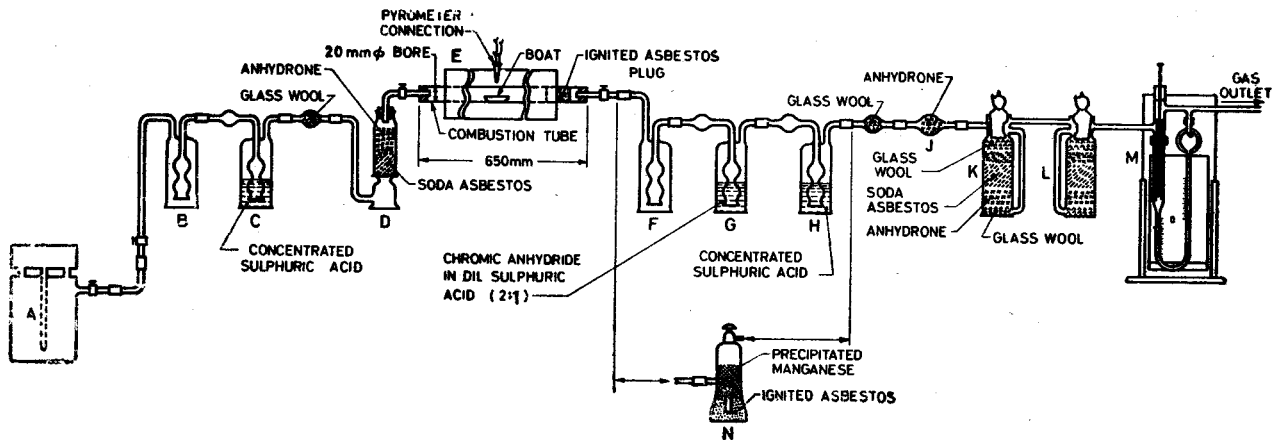


FIG. 1 APPARATUS FOR DETERMINATION OF CARBON (TOTAL) BY THE DIRECT COMBUSTION (GRAVIMETRIC) METHOD

any other suitable material, should be used. In the exit end of the tube an ignited asbestos plug 75 mm long is placed lightly to retain ferric oxide carried over by the gas stream.

11.2.5 Absorption Train — consists of an Arnold bottle (F) to prevent back suction, a gas absorption bottle (G) filled with chromic anhydride in dilute sulphuric acid 2:1 (v/v) to remove oxides of sulphur from the gas stream, a bottle (H) containing about 40 ml of concentrated sulphuric acid to remove most of the moisture from exit gas, the exit end of this bottle is filled with glass wool to remove sulphuric acid mist, one tube (J) containing 'anhydrone' or any other suitable desiccant and carbon dioxide absorption bottle (K) (80 ml capacity) containing a layer of soda asbestos or ascarite, which should be uniformly graded and evenly packed with a layer of anhydrone on the top, all connected in series with the exit end of the combustion furnace and the atmosphere. The fillings in (K) are held in position by small plugs of glass wool. A bottle (L) packed similarly to bottle (K) acts as a guard against atmospheric gases. Its exit end is attached to a flowmeter. Bottles (F), (G) and (H) may be replaced by a Midvale or Nesbitt bulb (N) containing precipitated manganese dioxide (*see* 11.3.1) over ignited asbestos.

11.3 Reagents

11.3.1 Precipitated Manganese Dioxide

- a) Dissolve 200 g of manganese sulphate ($\text{MnSO}_4, 4\text{H}_2\text{O}$) in 2500 ml of water, add concentrated ammonium hydroxide until ammoniacal, followed by 1000 ml of freshly prepared ammonium persulphate solution (22.5 percent) and heat to boiling. Boil for 10 minutes, adding more of concentrated ammonium hydroxide to maintain the ammoniacal condition. Further addition of solution of ammonium persulphate and ammonium hydroxide may be required until precipitation is judged to be complete.
- b) Discontinue boiling and allow the precipitate to settle; wash by decantation 6 to 8 times with 500 to 600 ml portions of water, allowing the precipitate to settle completely between washings. Finally, wash 2 to 3 times by decantation with 500 to 600 ml portions of dilute sulphuric acid. Transfer the precipitate to a Buchner funnel and wash with hot water until free from sulphates. Dry the residue at 105° to 110°C, grind to pass IS Sieves 85 to 70 (aperture 842 to 708 microns) and again dry thoroughly at 105° to 110°C.

11.3.2 Red Lead or Lead Chromate — solid.

11.4 Procedure

11.4.1 Heat the furnace to a temperature between 1200° to 1300°C. Determine the temperature by a standard thermocouple type pyrometer. Before starting an analysis, test the apparatus for any leaks. Pass a slow stream of oxygen for about 15 minutes through the whole train of the apparatus.

11.4.2 Weigh the carbon dioxide absorption tube every two minutes till constant weight is obtained.

11.4.3 Weigh accurately 2 g of the sample and transfer to a previously ignited porcelain boat with 3 g of red lead or lead chromate as flux, insert the boat quickly into the hot zone of the furnace, with rubber stopper replaced at the end of the combustion tube, wait for about a minute so that the boat attains the temperature of the furnace and pass a rapid stream (800 to 1000 ml per minute) of oxygen for 10 minutes. At the end of this period, remove the carbon dioxide absorption bulb. Cool in a desiccator and weigh quickly. The increase in weight represents the weight of carbon dioxide.

11.4.4 Carry out a blank determination at frequent intervals.

11.5 Calculation

$$\text{Carbon, percent} = \frac{(A-B) \times 27.29}{C}$$

where

A = weight in g of the carbon dioxide absorption tube after test,

B = weight in g of the carbon dioxide absorption tube in the blank, and

C = weight in g of the sample taken.

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